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BUREAU OF CHEMISTRY—CIRCULAR No. 13.

H. W. WILEY, Chief.

EXTRACTS FROM THE PROCEEDINGS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1903.

[An advance circular giving the recommendations of referees as adopted, the motions and appointments affecting the work of 1904.]

REPORTS OF COMMITTEES ON RECOMMENDATIONS OF REFEREES.

COMMITTEE A.—CHAIRMAN, B. L. HARTWELL.

(Phosphoric acid, potash, nitrogen, soils, ash, and insecticides.)

1. Phosphoric Acid.

No recommendations were made by the referee, and those made by the associate are practically covered by the recommendations on soils.

MOTION.

Mr. Wiley. I move that the referee on phosphoric acid be instructed to reconsider the question of the valuation of basic phosphatic slags, especially in regard to establishing a standard based on the total phosphoric acid and degree of fineness. The precipitated phosphates and Redonda phosphates are also to be considered.

2. Potash.

It is recommended—

1. That the question of the further trial of the modification requiring a slightly acid solution and neutralization with sodium hydroxid be left to the discretion of the referee.

2. That the association take some action to ascertain whether the members desire to determine moisture in potash salts by drying at a stated temperature or by ignition, and that the study of the method thought most desirable be continued with a view to obtaining more concordant results for water in potash salts.

(The committee, without action, presented the above to the association, which referred the whole question to the referee for 1904.)

3. NITROGEN.

DETERMINATION OF NITROGEN.

It is recommended—

1. That in the provisional neutral permanganate method the first two lines be changed to read as follows:

Into a 300 cc low form Griffin beaker weigh 2 grams of the sample, if from a mixed fertilizer; if from concentrated material use a quantity containing approximately 0.075 gram of nitrogen.[a]

2. That the study of methods for the determination of available organic nitrogen be continued.

SEPARATION OF NITROGENOUS BODIES.

Milk and cheese proteids.

It is recommended—

1. That the method given [in the referee's report] for the separation and determination of casein monolactate and casein dilactate be adopted as a provisional method.

Casein monolactate and casein dilactate.

1. *Determination of casein monolactate in milk.*—Casein monolactate in milk coagulates readily at 40° C. Hence, in a milk containing only casein and casein monolactate, the monolactate can be separated by heating the milk to about 40° C., filtering the precipitate formed, washing and determining the nitrogen in the precipitate. Ten grams of milk diluted with 90 cc of water give good results.

2. *Separation of casein monolactate and casein dilactate.*—Casein dilactate coagulates completely at 40° C. and below. In milk containing casein monolactate and dilactate, heat 10 grams of milk diluted with 90 cc of water to 40° C., and these two salts precipitate and are separated from the milk casein by filtration. The washed precipitate is then treated with 100 cc of a 5 per cent solution of sodium chlorid, and the whole heated to 55° C., with frequent agitation for two hours. The process is facilitated somewhat by the presence of pure quartz sand. The casein monolactate goes into solution and is separated from the casein dilactate by filtration and washing.

3. *Separation and determination of casein, casein monolactate, and casein dilactate in milk.*—(a) The total amount of nitrogen precipitated by acid is determined by the official method prescribed for determining casein in milk.

(b) Heat 10 grams of milk diluted with 90 cc of water to 40° C. for fifteen or twenty minutes, filter the precipitate formed, and wash with distilled water. Transfer the precipitate to a small Erlenmeyer flask, provided with a stopper, treat with 100 cc of a 5 per cent solution of sodium chlorid and heat at 55° C. for two hours, with frequent agitation. Filter the mixture, wash the remaining precipitate with water, and determine the nitrogen in both the precipitate and filtrate. The nitrogen in the precipitate represents casein dilactate; that in the filtrate, casein monolactate. The sum of these two substracted from the total nitrogen found by precipitation with acid gives the amount of nitrogen as casein.

Vegetable proteids.

1. That Mr. Harcourt's report on vegetable proteids be included in the printed proceedings.

Meat proteids.

1. That the following methods be given further trial by the association:

Phosphotungstic acid alone in hot and cold solutions.

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[a] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 67, p. 77, or Bul. No. 73, p. 49.

Phosphotungstic acid, followed by bromin.
Phosphotungstic acid, followed by zinc sulphate.
Tannin salts, followed by zinc sulphate.

2. That the method for precipitation by bromin alone, and by bromin in the filtrate from zinc sulphate, be discontinued.

4. SOILS.

It is recommended–

1. That in the continuation of the work on methods for available phosphoric acid the referee compare greater concentrations than N/200 acid.

2. That the recommendation calling for a continuation of the work on the solubility of phosphoric acid and potash in the different soil layers and for an investigation of methods for determining total phosphoric acid be left to the discretion of the referee.

3. That the following provisional method for the analysis of the water-soluble portion of alkali soils be given further study before its adoption as a provisional method.

PROVISIONAL METHOD FOR THE DETERMINATION OF WATER SOLUBLE CONSTITUENTS OF ALKALI SOILS.

(a) *Preparation of solution.*

Place 25 grams of soil in a graduated 500 cc flask, fill to the mark with distilled water, allow to stand 20 to 24 hours, shaking frequently and filter through a dry filter or if clear siphon off the supernatant liquid.

(b) *Determination of total carbonates.*

Titrate an aliquot portion of this filtrate with N/20 sulphuric acid, using a few drops only of methylorange as indicator, and be very careful not to run over the end point. From the number of cubic centimeters of acid required calculate the total CO_2 as carbonates.

(c) *Determination of chlorin.*

To the aliquot portion used for the titration of carbonates add a few drops more of N/20 acid, then a little potassium chromate and titrate with N/10 silver nitrate and calculate to chlorin (cc N/10 silver nitrate multiplied by 0.003518 = the number of grams of chlorin).

(d) *Determination of sulphuric acid.*

An aliquot portion of the original filtrate is made slightly acid with hydrochloric acid, and if a precipitate forms the solution is filtrated and the filter washed free of sulphates. To the boiling solution add slowly a large excess of barium chlorid solution and finish the determination as in 4 (j) [a]. In solutions containing large amounts of carbonates the ignited barium sulphate should be treated with hydrofluoric acid and a few drops of sulphuric acid ignited and again weighed.

(e) *Determination of bases.*

If the original filtrate is turbid, as it is apt to be when the soil contains carbonates and but little of other salts, it may be cleared by adding 5 cc of thick alumina cream to 200 or 300 cc and filtering through a dry filter. In using portions of this filtrate for the determinations correction should be made for the added alumina cream. To aliquot portions of the solution add hydrochloric acid in excess, and in cases where silica may be present separate in the usual way and determine the bases as in 4 (b), (c), (d), and (e).[a]

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[a] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 46, Methods of Analysis, p. 75.

(f) *Determination of phosphoric acid.*

Add an excess of nitric acid to an aliquot portion of the original solution. Make nearly neutral with ammonia, precipitate with ammonium molybdate, finishing the determination as in 4 (g).[b]

4. That on page 72, under 4 (a), acid digestion of the soil, for the words "and again evaporate to complete dryness" substitute the following: Filter, wash free of chlorids, and again evaporate to complete dryness as before.

5. That on page 74, under 4 (g), determination of phosphoric acid, the official method given be marked "(a)" and the following optional provisional method be inserted as (b):

(b) *Optional provisional method:* Proceed as in (a) "until all the phosphoric acid is precipitated," and then finish the determination as follows:

After standing for three hours at a temperature not above 50°, filter on a small filter, wash with water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by one drop of standard alkali. Place the filter and precipitate in the beaker and dissolve in standard alkali, add a few drops of phenolphthalein solution and titrate with standard acid, 1 cc of which equals 0.0005 gram of phosphoric acid (P_2O_5).

6. That on page 74, 4 (h), the word "available" be omitted from the heading "Provisional method for the determination of available phosphoric acid."

7. That on page 76, paragraph 10, determination of humus, the tenth line, the sentence beginning, "The supernatant liquid is filtered, etc.," shall be changed to read as follows: "The supernatant liquid is filtered, and the filtrate must be perfectly clear and free from turbidity; evaporate an aliquot portion, dry at 100°, and weigh."

8. That the retiring referee be instructed to make such verbal corrections in the methods as these changes will involve, and submit them to the secretary for approval and publication.

RESOLUTIONS.

W. A. WITHERS. *Resolved,* That the referee on soils be requested to test, if possible, the value of proposed methods for comparing the ability of different soils to support the growth of nitrifying organisms when the conditions are identical as to temperature, moisture, number of germs, etc.

C. L. PENNY. *Resolved,* That the referee on soils be requested to make a thorough examination of the methods described in Bulletin 22 of the Bureau of Soils, for the determination of available plant food.

5. ASH.

It is recommended—

1. That the method for the determination of sulphates, as described on page 25 of the Proceedings of the Nineteenth Annual Convention of this association, 1902,[c] be printed as a provisional method.

2. That the peroxid method of determining sulphates be subjected to further study, with a view to substituting it for the nitric acid method.

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[a] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 46, Methods of Analysis, pp. 72, 73.

[b] Ibid., p. 74.

[c] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 73.

6. INSECTICIDES.

It is recommended—

1. That Method I for total arsenious oxid in Paris green be adopted as an official method, this being a repetition of last year's recommendation.

Adopted as an official method.

2. That the four modifications of the Avery-Beans method for total arsenic in Paris green be tested to determine which is the best modification to be adopted as an optional official method.

3. That the electrolytic method be adopted as the official method for determining copper in Paris green, and in copper carbonate, and that the volumetric methods be further tested, paying particular attention to the remarks of Messrs. Davidson, McBryde, and Allen upon this subject.[a] (Recommended for the first time.)

4. That an attempt be made to so change the procedure in the analysis of London purple that some of the organic matter may be removed. The remarks of Messrs. Davidson and McBryde are of value in this connection.[a]

5. That the volumetric silver nitrate method be adopted as the official method of determining cyanogen in potassium cyanid and that an N/20 instead of N/10 solution of silver nitrate be used. (Recommended for the first time.)

6. That the methods of examination of soda lye be further tested and compared with gravimetric methods and that the points brought out by Messrs. Davidson and McBryde be given consideration.[a]

7. That the Kissling method for determining nicotin be adopted as an official method. (Recommended for the first time.)

8. That special attention be given to the methods for determining formaldehyde in order to avoid needless repetition of work.

MOTION.

A motion made by Mr. Bigelow and amended by Mr. Haywood was passed, referring to the referee on insecticides the study of methods for the examination of disinfectants.

COMMITTEE B.—CHAIRMAN, G. E. PATRICK.

(Dairy products, foods and feeding stuffs, sugar and tannin.)

1. DAIRY PRODUCTS.

It is recommended—

1. That the referee on dairy products be requested to continue the investigation of the effects of preservatives upon the albumin of milk.

2. FOODS AND FEEDING STUFFS.

It is recommended—

1. That the referee be requested to study the composition of the ether extract obtained after digesting with pepsin the residue obtained by the official method of fat extraction.

2. That the modified König method for the determination of crude fiber be further investigated, especially to ascertain whether the glycerol-acid mixture hydrolyzes any of the true fiber.

3. SUGAR.

It is recommended—

1. That the associate referee on chemical methods be instructed to cooperate with the International Association for the Unification of Sugar Methods to the end that a uniform method for the determination of reducing sugars be obtained.

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[a] See Proceedings of the Twentieth Annual Convention A. O. A. C., 1903, now in preparation.

2. That an associate referee on methods of analysis of molasses be appointed.

3. That the following methods and changes in methods be adopted:

a. Changes in the official methods given in paragraph 1, page 56, and paragraph 2, page 57, of the Proceedings of this association for 1902.[a]

Adopted as official.

b. The provisional method given in paragraph (f), page 57, of the Proceedings of the association for 1902.

c. Under "Optical methods for the determination of sucrose" omit Clerget's method and Creydt's method (pp. 39 and 40 of Bul. No. 46, Methods of Analysis) and substitute therefor the German official methods for sucrose and raffinose as the official methods of this association. These methods are given on page 57 of the Proceedings of the association for 1902, but in the twenty-fifth line from the top of the page it is recommended to change the words "Dissolve 13.024 grams of the substance in 75 cc of water," so as to read, "Take 50 cc of the clarified solution freed from lead, add 25 cc of water."

Adopted as official.

d. As an alternate official method for inversion without the application of heat the following is recommended:

> Take 50 cc of the clarified solution freed from lead, add 5 cc of hydrochloric acid containing 38.8 per cent of acid, set aside during a period of 24 hours at a temperature not below 20° C.; or if the temperature be above 25° C. set aside for 10 hours. Make up to 100 cc at 20° C. and polarize.

Adopted as official.

e. As provisional methods for the analysis of sugar beets:

Scheibler's alcoholic method and Pellet's aqueous method (p. 58, Proceedings for 1902).

f. As provisional, the methods selected by the international committee for unifying methods of sugar analysis (pp. 58, 59, Proceedings for 1902, pars. 1 to 9, inclusive).

4. That the referees on sugar be requested to continue investigations along the same lines as during the past year.

TANNIN.

The following recommendations were not received until after Committee B had finished its work, and they were referred by the association to the referee for 1904:

It is recommended—

1. That the last sentence of Paragraph II, Quantity of Material (Bul. 46, Methods of Analysis, p. 79), be changed to read as follows:

> In the case of extract, weigh in a weighing bottle, fitted with a ground-glass stopper, such quantity, etc.

2. That the last sentence of Paragraph V, Soluble Solids (Bul. 46, Methods of Analysis, p. 80), be changed to read as follows:

> To guard against loss by evaporation, the flask receiving the filtered liquor shall be stoppered with a rubber stopper and the funnel stem passed through the perforation. The funnel shall be covered with a glass plate having an orifice only sufficiently large to admit the air required by the displacement of the liquor.

MOTION.

A motion made by Mr. Stilwell, and amended by Mr. Veitch, to the effect that the time of extraction of spent tan bark be fixed at 10 hours at steam heat, was referred to Committee B, but no action was taken.

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[a] U. S. Dept. of Agr., Bureau of Chemistry, Bul. No. 73.

COMMITTEE C.—CHAIRMAN, A. E. LEACH.

(Food adulteration.)

1. Distilled Liquors.

On page 96 of Bulletin No. 65,[a] under Determination of Alcohol, substitute for the first sentence the following: "Weigh or measure (at 15.6° C.) in a distilling flask 20 to 25 cc of the sample, dilute with 100 cc of water, and proceed as directed on page 82."

On the same page, under Determination of Extract, strike out the method there given and insert the following: "Weigh or measure (at 15.6° C.) 100 cc of the sample, evaporate nearly to dryness on the water bath, then transfer to a water oven, and dry at the temperature of boiling water."

On the same page, under Determination of Acidity, strike out the method given and insert the following:

Titrate 100 cc (or 50 cc diluted to 100 cc if the sample is dark in color) with decinormal barium hydrate, using phenolphthalein as indicator. The number of cubic centimeters employed is multiplied by 0.006 for the acidity expressed in grams of acetic acid per 100 cc.

On the same page, under the Determination of Fusel Oil, strike out the third paragraph and substitute the following:

Add a small quantity of alkali to 200 cc of the sample under consideration and distill slowly, till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again until the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume[b] (sp. gr. 0.96541 at 15.6°).

On page 98 strike out the method given under Determination of Ethereal Salts and substitute the following:

Neutralize the residue left after distillation in the fusel oil determination with N/10 H_2SO_4 and add an excess of 10 cc of the acid. Let stand five minutes and make up to 200 cc. Titrate 2 portions of 25 cc each, using as indicators methyl orange in the first and phenolphthalein in the second. The difference gives the amount of alkali necessary to neutralize the organic acids in 25 cc of the sample. By subtracting from this figure the number of cubic centimeters of alkali required for the free acids, and multiplying the result by 0.0088, the number of grams of ethereal salts (calculated as ethyl acetate) in 25 cc of the sample is determined.

2. Dairy Products.

On page 36, under Detection of Formaldehyde, designate the method there given as (a) *Hehner's method,* and insert the following method, lettered (b):

(b) *The hydrochloric acid and ferric chlorid method.*[c]—To 10 cc of milk in a porcelain casserole add an equal volume of concentrated hydrochloric acid containing 1 cc of 10 per cent ferric chlorid solution to each 500 cc of acid. Heat nearly to the boiling point over the free flame, holding the casserole by the handle and giving it a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

Insert also as (c) the phloroglucin method as described at the foot of page 72, to the end of the first paragraph on page 73.

On page 36, after section 7, "Detection of Borax and Boric Acid," insert the following:

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[a] U. S. Dept. Agr., Bureau of Chemistry, Provisional Methods for the Analysis of Foods.

[b] Refers to footnote "*b*" on p. 96 of Bul. No. 65.

[c] An. Rep. Mass. State Board of Health, 1897, p. 558. Food and Drug Reprint, p. 20.

8. DETECTION OF BENZOIC ACID.[a]

Add 5 cc of dilute hydrochloric acid to 50 cc of the milk in a flask and shake to curdle. Then add 150 cc of ether, cork the flask, and shake well. Break up the emulsion which forms by aid of a centrifuge, or if the latter is not available extract the curdled milk by gently shaking with successive portions of ether, avoiding the formation of an emulsion.[b] Transfer the ether extract (evaporated to small volume if large in bulk) to a separatory funnel and separate the benzoic acid from the fat by shaking out with dilute ammonia, which takes out the former as ammonium benzoate. Evaporate the ammonia solution in a dish over the water bath till all free ammonia has disappeared, but before getting to dryness add a few drops of ferric chlorid reagent. The characteristic flesh-colored precipitate indicates benzoic acid. Care should be taken not to add the ferric chlorid till all the ammonia has been driven off, otherwise a precipitate of ferric hydrate is formed.

9. DETECTION OF SALICYLIC ACID.

Proceed exactly as directed for benzoic acid. On applying the ferric chlorid to the solution after evaporation of the ammonia, the well-known violet color indicates salicylic acid, when present.[c]

On page 36, "Detection of Foreign Colors" should be renumbered as "10." On page 39, change heading 8 to "Detection of Foreign Colors" inserting "(a) *Cornwall's method* [c]" before the method as there given.

On page 39, following Cornwall's method, insert the following:

(b) *Method of the Massachusetts Board of Health for annato.*—Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm, dilute sodium hydroxid, and after stirring pour the mixture while warm upon a wet filter, using to advantage a hot funnel. If annato is present, the filter will absorb the color so that when the fat is washed off by a gentle stream of water the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color. If, after drying the filter, the color turns pink on application of a drop of stannous chlorid solution the presence of annato is assured.

(c) *Geisler's method for azo colors.*[d]—A few drops of the clarified fat are spread out on a porcelain surface and a pinch of fuller's earth added. In the presence of various azo dyes a pink to violet-red coloration will be produced in a few minutes. Some varieties of the fuller's earth react much more readily than others with azo colors.

(d) *Low's method for azo colors.*[e]—A small amount of the material to be tested is melted in a test tube, an equal volume of a mixture of one part of concentrated sulphuric acid and four parts of glacial acetic acid is added and the tube is heated nearly to the boiling point, the contents being thoroughly mixed by shaking. The tube is then set aside and after the acid solution has settled out it will be found to be colored wine-red in the presence of azo color, while with pure butter fat comparatively no color will be produced.

(e) *Doolittle's method for azo dyes and annato.*[f]—The melted sample is first filtered. Two test tubes are taken and into each are poured about 2 grams of the filtered fat which is dissolved in ether. Into one test tube 1 or 2 cc of dilute hydrochloric acid are poured and into the other about the same volume of dilute potassium hydroxid solution. Both tubes are well shaken and allowed to stand. In the presence of azo dye the test tube to which the acid has been added will show a pink to wine-red coloration while the potash solution in the other tube will show no color. If annato has been used, on the other hand, the potash solution will be colored yellow while no color will be apparent in the acid solution.

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[a] Leach. An. Rep. Mass. State Board of Health, 1902. Food and Drug Reprint, p. 23.

[b] A volume of ether largely in excess over that of the curdled milk has been found to be less apt to form an obstinate emulsion.

[c] These methods for salicylic and benzoic acids while especially applied to milk, from which the ether extracts both fat and preservative, are useful also with modifications for many other food products. The extraction of the ether solution with dilute ammonia, whereby the preservative is removed, permits the subsequent recovery of the ether by distillation.

[d] Jour. Am. Chem. Soc., 1898, **20**: 110.

[e] Jour. Am. Chem. Soc., **20**: 889.

[f] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 65, p. 152.

Also on page 39 strike out section 9, Detection of Aniline Colors, and in its place insert the following:

9. Detection of Boric Acid in Butter.

Melt about 25 grams of the sample on the water-bath, pour off the fat from the aqueous solution that settles to the bottom of the container, acidify the aqueous solution slightly with hydrochloric acid and test in the usual manner with tumeric paper for boric acid.

On page 40, under Determination of Fat, insert the following heading for the method there given: "(a) *Official method.*[e]"

On page 41, at the end of the first paragraph, insert the following:

(b) *Lythgoe's modification of the Babcock method.*—Weigh accurately about 6 grams of the sample in a tared beaker. Add 10 cc of boiling water and stir with a rod till the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonia to aid in the softening and emulsionizing, and keep the beaker in hot water till the emulsion is tolerably complete and free from lumps.

If the sample is a full cream cheese a Babcock cream bottle is employed. The contents of the beaker, after cooling, are transferred to the test bottle as follows: Add to the beaker about half of the 17.6 cc of sulphuric acid regularly used for the test, stir with a rod and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally proceed as in the Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample taken to obtain the per cent of fat.

On page 41, at the end of the methods for dairy products, insert the following:

(C) Condensed Milk.

Preparation of the sample.—Mix thoroughly by transferring the contents of the can to a large evaporating dish and working it with a pestle until homogeneous. Weigh 40 grams of the mixed sample in a 100 cc flask, or transfer thereto by washing, and make up to the mark with water.

Total solids.—Dilute a measured portion of the above 40 per cent solution with an equal amount of water, and transfer by a pipette 5 cc of the diluted mixture, corresponding to 1 gram of the condensed milk, into a tared platinum dish, which is allowed to remain in contact with the live steam of a water-bath for at least two hours after the last traces of water have been evaporated to leave an apparently dry residue. Transfer to a desiccator, cool, and weigh.

Ash.—Carefully ignite the residue from the total solids, cool, and weigh.

Fat.[a]—Measure 15 cc of the above 40 per cent solution, corresponding to 6 grams of the condensed milk, into a Babcock test bottle. Fill nearly to the neck with water and add 4 cc of Fehling's copper solution, shake thoroughly and rapidly, separating the precipitated proteids and fat by means of a centrifuge,[b] or the precipitate may be allowed to settle of itself, which it does more quickly in the cold. Withdraw the supernatant sugar-containing liquid by means of a small stemmed pipette with a wisp of wet absorbent cotton twisted over the bottom to serve as a filter. Wipe off the cotton into the bottle on withdrawing the pipette. Give the precipitated proteids and fat two additional washings as above by shaking with water, separating the precipitate, and removing the washings with the pipette. If the precipitate is caked hard after centrifuging, use a stiff platinum wire as a stirrer. Finally add water to an approximate volume of 17.5 cc and 17.5 cc of sulphuric acid, and continue the test as in the Babcock process of milk testing, multiplying the reading by 3 for the percentage of fat in the sample.

Proteids.—Dilute 5 cc of the 40 per cent solution, corresponding to 2 grams of the sample, to about 40 cc and add 0.6 cc of Fehling's copper solution. Nearly neutralize with sodium hydroxid, stopping just short of alkalinity. Pass through a weighed filter paper, wash, dry at 100° C., and weigh. Burn the precipitate in a porcelain crucible, the difference between the weight of the dry precipitate and the weight of the ash being the weight of the proteids and fat. Expressing this in percentage and deducting the per cent of fat previously obtained, the result is the per cent of proteids.

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[a] Leach, Jour. Am. Chem. Soc., 1900, **22**: 589.

[b] While the steam-driven centrifuge may be used for this, it is better to centrifuge in the cold, since the heat of the steam-driven machine cakes the precipitate so that it is harder to wash.

Milk sugar.—Make up the filtrate and washings from the previous operation to 100 cc and determine the lactose either gravimetrically or volumetrically in this solution. If the volumetric method is used and the solution is of the exact strength directed above, milk sugar may be calculated as follows:

$$\frac{100 \times 0.067}{S \times 0.02} = L$$

wherein L is the per cent of lactose, and S the number of cubic centimeters of milk solution prepared as above necessary to reduce 10 cc of Fehling's solution.

Cane sugar.—Determine by difference, deducting the milk solids (milk sugar + proteids + fat + ash) from the total solids.

3. Saccharine Products.

On page 48, under Determination of Commercial Glucose in Molasses Sirups and Honey make the following changes:

At the end of the second paragraph insert "Express results in terms of commercial glucose polarizing at 175°."

Cancel the third paragraph and substitute the following:

In honey, which is composed largely of invert sugar, much more accurate results are attained by polarizing at a temperature of 87° in a water-jacketed tube an inverted, half normal solution of the sample prepared as follows: Weigh out one-half the normal weight of the sample (13.024 grams) in a 100 cc graduated flask, dissolve in about 70 cc of water, and add 7 cc of concentrated hydrochloric acid; then heat to 68° C. and cool in the usual manner. After inversion, add a few drops of phenolphthalein and enough sodium hydroxid to neutralize. Discharge the pink color with a few drops of dilute hydrochloric acid, cool again, add from 5 to 10 cc of alumina cream and make up to the mark. Multiply by 2 the reading at 87° in the 200 mg tube; divide the result by the factor 163 to express the glucose in terms of glucose polarizing at 175°.

Strike out footnote b, in the fourth paragraph.

4. Edible Oils and Fats.

On page 26, before heading 5, Determination of Saponification Number and Soluble and Insoluble Acids, insert the Hanus method as a provisional optional method. This method will be printed in full in the Proceedings for 1903 now in preparation; the principal points in which it differs from the Hübl method are as follows:

Dissolve 13.2 grams of iodin in 1,000 cc glacial acetic acid (showing no reduction with bichromate and H_2SO_4), and add enough bromin to double the halogen content determined by titration. Three cc of bromin is about the proper amount. The iodin may be dissolved by the aid of heat, but the solution should be cold when bromin is added. The solution is used in the same manner as in the Hübl method except that an excess of 70 per cent of the iodin is allowed; 10 cc of the KI solution is added instead of 20 cc and the time of reaction is shortened to thirty minutes. When the KI solution is added, shake the mixture thoroughly before adding the water.

On page 32, under heading 16, Halphen reaction for cotton-seed oil, third line, insert before the word "brine" the word "saturated," and for "fifteen minutes" substitute "an hour to 2 hours."

On page 33, under heading 18, Renard's Test for Peanut Oil, etc., substitute the method to be printed in full in the Proceedings for 1903, now in preparation. This method is essentially the same as that given on page 33, except that 20 grams of fat are taken for analysis with the proportional increase of reagents rendered necessary by the larger amount of fat. An additional washing is also provided for in the revised method.

In Bulletin 46, page 49, under Determination of Refractive Index, second paragraph, line 3, change the factor 0.000176 to 0.000365, in accordance with the more correct factor given in Bulletin 65. Also cancel the example following and substitute the example given on page 22 of Bulletin 65, under Determination of Index of Refraction. (Recommended for the first time.)

5. Spices.

On page 58 of Bulletin 65, under Determination of Starch by the Diastase Method, fifteenth line, strike out the words "copper reduced by" and insert therefor "dextrin resulting from the inversion of."

6. Fruits and Fruit Products.

The following changes in the provisional methods as given in Bulletin 65 are recommended:

(1) On page 78, under heading 12, Polarization, substitute the method given on page 57 of the Proceedings for 1902, under Determination of sucrose in absence of raffinose.

(2) On page 78, section 13, b, line 8, change "1 per cent" to "0.50 per cent."

(3) On page 78, section 14, lines 8 and 9, for "use Allihn's method for the determination (p. 49) [a]," substitute "Use the official method for the determination of invert sugar as given in Bul. 46, p. 33 (c) (1) (a) and (b)."

(4) On page 78, strike out section 15, Determination of Dextrin, and substitute therefor Determination of Glucose, referring to method given on page 48, section 12.

7. Vinegar.

On page 65 of Bulletin 65, following section 13, Detection of Coloring Matters, insert Crampton and Simon's method for the detection of caramel as follows:[a]

Add 25 grams of fuller's earth to 50 cc of the vinegar under examination, beat the mixture up in a beaker and let it stand covered half an hour at room temperature, then filter. The determination of the figure representing the color is made with the tintometer upon the liquid before and after treatment and the difference between the two results gives the percentage of color absorbed.

8. Cocoa and Cocoa Products.

The report submitted is to be printed separately and distributed to the associate referees on food adulteration for criticism before it is printed in the Proceedings, in accordance with the plan previously pursued.

RESOLUTIONS AND MOTIONS BEARING ON THE WORK OF REFEREES AND COMMITTEES FOR 1904.

Cooperation with the Bureau of Standards.

Resolved, That the Bureau of Standards be invited to cooperate with the referees of the association in so far as possible in establishing the accuracy of standard methods of analysis.

(The following letter has been received from Director Stratton, and the referees are, therefore, requested to ask the Bureau of Standards to collaborate in their work.—H. W. W.)

December 8, 1904.

Dear Sir: I beg to acknowledge the receipt of your letter of December 4, transmitting resolutions passed by the Association of Official Agricultural Chemists inviting the Bureau of Standards to cooperate with the association in establishing the accuracy of standard methods of analysis. In reply I would state that the Bureau of Standards will be pleased to cooperate with the association in every way consistent with its functions. This question is one in which the Bureau, and especially our chemical section, is very much interested.

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[a] J. Amer. Chem. Soc., 1899, **21**: 355.

Respectfully,

S. W. STRATTON, *Director.*

COOPERATION WITH THE AMERICAN CHEMICAL SOCIETY.

Resolved, That the chair be instructed to appoint a committee from the Association to cooperate with the Committee of the American Chemical Society in the matter of testing chemicals for purity.

The president appointed the following committee: Chairman, L. F. Kebler, Bureau of Chemistry, Department of Agriculture, Washington, D. C.; A. L. Winton, Agricultural Experiment Station, New Haven, Conn.; B. W. Kilgore, Department of Agriculture, Raleigh, N. C.

REFEREE ON DRUGS.

Moved, by H. W. Wiley, that a referee on the analysis of medicinal plants and drugs made therefrom be appointed.

FERTILIZER LEGISLATION.

Moved, by H. W. Wiley, that the Committee on Fertilizer Legislation be instructed to confer with manufacturers and distributers of fertilizer materials and with such other persons as may be interested in the matter and report at the next meeting the form that national legislation should take with a view to preparing a tentative bill and urging its enactment into a law.

CONVERSION TABLES.

Moved, by H. W. Wiley, that the president be requested to appoint a committee of three, of which Mr. H. B. McDonnell shall be the chairman, to consider the matter of publishing in the Proceedings of the association calculation tables, and that the committee be authorized to submit its report to the secretary of the association with recommendation.

(C. B. Williams, Department of Agriculture, Raleigh, N. C., and B. B. Ross, Agricultural Experiment Station, Auburn, Ala., were appointed.)

PLACE OF MEETING, 1904.

Moved, by L. L. Van Slyke, that the Executive Committee be authorized to select a place of meeting for 1904 and that they be requested to consider the feasibility of holding that meeting in St. Louis.

(The Executive Committee has authorized the secretary to call the meeting at St. Louis as suggested.—H. W. W.)

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1904.

President.

Mr. M. E. Jaffa, Berkeley, Cal.

Vice-President.

Mr. C. L. Penny, Newark, Del.

Secretary.

Mr. H. W. Wiley, Washington, D. C.

Additional members of Executive Committee.

Mr. W. P. Headden, Fort Collins, Colo.
Mr. W. R. Perkins, Agricultural College, Miss.

Referees.

Phosphoric acid: C. B. Williams, Raleigh, N. C.
Nitrogen:
    Determination of nitrogen: C. H. Jones, Burlington, Vt.
    Separation of nitrogenous bodies: E. B. Hart, Geneva, N. Y. (milk and cheese proteids).
Potash: F. B. Carpenter, Richmond, Va.
Soils: C. G. Hopkins, Urbana, Ill.
Dairy products: G. E. Patrick, Washington, D. C.
Foods and feeding stuffs: J. O. LaBach, Lexington, Ky.
Food adulteration: W. D. Bigelow, Washington, D. C.
Sugar: L. S. Munson, Washington, D. C.
Tannin: George A. Kerr, Damascus, Va.
Insecticides: B. H. Smith, Washington, D. C.
Ash: R. W. Thatcher, Pullman, Wash.
Medicinal plants and drugs: L. F. Kebler, Washington, D. C.

Associate referees.

Phosphoric acid: F. P. Veitch, Washington, D. C.
Nitrogen:
    Determination of nitrogen: F. A. Urner, Geneva, N. Y.
    Separation of nitrogenous bodies:
        Meat proteids: W. D. Bigelow, Washington, D. C.
        Vegetable proteids: J. S. Chamberlain, Washington, D. C.
Potash: G. S. Fraps, College Station, Tex.
Soils: R. H. Loughridge, Berkeley, Cal.
Dairy products: F. W. Woll, Madison, Wis.
Foods and feeding stuffs: J. K. Haywood, Washington, D. C.

Food adulteration:

(1) Colors: W. G. Berry, Appraiser's Office, New York, N. Y.
(2) Saccharine products, including confectionery: E. B. Kenrick, Winnepeg, Manitoba, Canada.
(3) Fruit products: E. M. Chace, Washington, D. C.
(4) Wine: G. E. Colby, Berkeley, Cal.
(5) Beer: H. E. Barnard, Concord, N. H.
(6) Distilled liquors: C. A. Crampton, Washington, D. C.
(7) Vinegar: Richard Fischer, Madison, Wis.
(8) Flavoring extracts: R. E. Doolittle, Lansing, Mich.
(9) Spices: A. L. Winton, New Haven, Conn.
(10) Baking powder and baking chemicals: R. O. Brooks, Trenton, N. J.
(11) Meat and fish: M. E. Jaffa, Berkeley, Cal.
(12) Fats and oils: L. M. Tolman, Washington, D. C.
(13) Dairy products: A. E. Leach, Boston, Mass.
(14) Cereal products: A. McGill, Ottawa, Canada.
(15) Infants and invalids' foods: H. W. Wiley, Washington, D. C.
(16) Vegetables: F. W. Bedford, St. Paul, Minn.
(17) Condiments other than spices: J. D. Hird, Washington, D. C.
(18) Cocoa and cocoa products: E. N. Eaton, Chicago, Ill.
(19) Tea and coffee: H. C. Lythgoe, Boston, Mass.
(20) Preservatives: W. D. Bigelow, Washington, D. C.

Sugar:

Molasses methods: H. E. Sawyer, Boston, Mass.
Special analytical methods: C. A. Browne, jr., Audubon Park, La.

Tannin: H. C. Reed, Stamford, Conn.
Insecticides: S. Avery, Lincoln, Nebr.
Ash: F. T. Shutt, Ottawa, Canada.

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